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Chemical Study of Synthesized Organic Compounds from (Shiff Bases-Sugars)

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ABSTRACT

In this research Schiff bases-Sugars used as starting material in synthesis of new organic compounds (azitidine, tetrazole, oxazepine, diazepine, thio compound, amide compound) which some of them have cyclic structure(four, five, seven)-membered ring. All synthesized compounds[1-10] characterized by chemical techniques (FT-IR,H^1-NMR, C.H.N-analysis) and melting points with physical properties.

Keywords: sugars, schiff base, imine-sugars.

INTRODUCTION

Carbohydrate are a major class of naturally occurring organic compounds, which involves only Two functional groups-ketone or aldehyde carbonyls and alcohol hydroxyl groups. During the Past few years carbohydrates have received increasing attention as stereo differentiating auxiliaries in stereo selective synthesis[1,2]. The presence of acarbohydrate moity side chain in any synthesized compound may overcome the Frequently observed water insolubility problem. On the other hand,the incorporation of imine-mono saccahrides compound with other Compounds such as sodium azide or chloro acetyl chloride…etc, to produce fused rings and open rings compounds which was known to possess various pharmacological activities like antibacterial, analegesic, anti inflammatory, anticonvulsant, antimicrobial activities[3-5]. The hetero cyclic compounds bearing sugars in their structure have many applications in Biological science, and most of imine compounds bearing mono or bi cycles have chemical[(6)]and Biological importance[7-11].

EXPERIMENTAL

All chemical used from merck and BDH-company, and measurements were carried out by: FT-IR Spectra-Shimadzue (8300), KBr-disk in Co.S.Q-Iraq., H-NMR

spectra and (C.H.N)-analyzer in Jordan., melting points in electrothermal 9300, melting point engineering LTD,U.K.

Synthesis of compound[1]

According procedure[12], equi molar mixture(0.1 mole) of 2-Amino thiazol (10 gm) with (15gm) of xylose sugar were reacted Under refluxing for (3hrs) in presence of glacial acetic acid(drops) and absolute ethanol as Solvent with stirre by used mechanical stirre,the precipitate filtered and dried, recrystallized From absolute ethanol to give 82% from imine-xylose named compound[1].

Synthesis of compounds[2-4]

A mixture of equi molar (0.01 mole) of (2.3gm) compound[1] with one of {(1.13gm of chloro acetyl Chloride), (0.65gm of sodium azide),(0.98gm of maleic anhydride)} respectively were dissolved in Dioxane with stirre,then the mixture was refluxed for(8-10)hrs, then theprecipitate filtered and Dried,recrystallize to produce (azitidine 86%,tetrazole 84%, oxazepine[11] 86%) from Compounds[2-4]respectively.

Synthesis of compound[5]

According procedure[13], equi molar mixture (0.01mole) of (3.3gm) compound[4] with (0.33gm) of amine hydroxyl were Refluxed for (7)hrs in presence of dioxane to produce 88%from diazepine cycle named Compound[5].

Synthesis of compound[6]

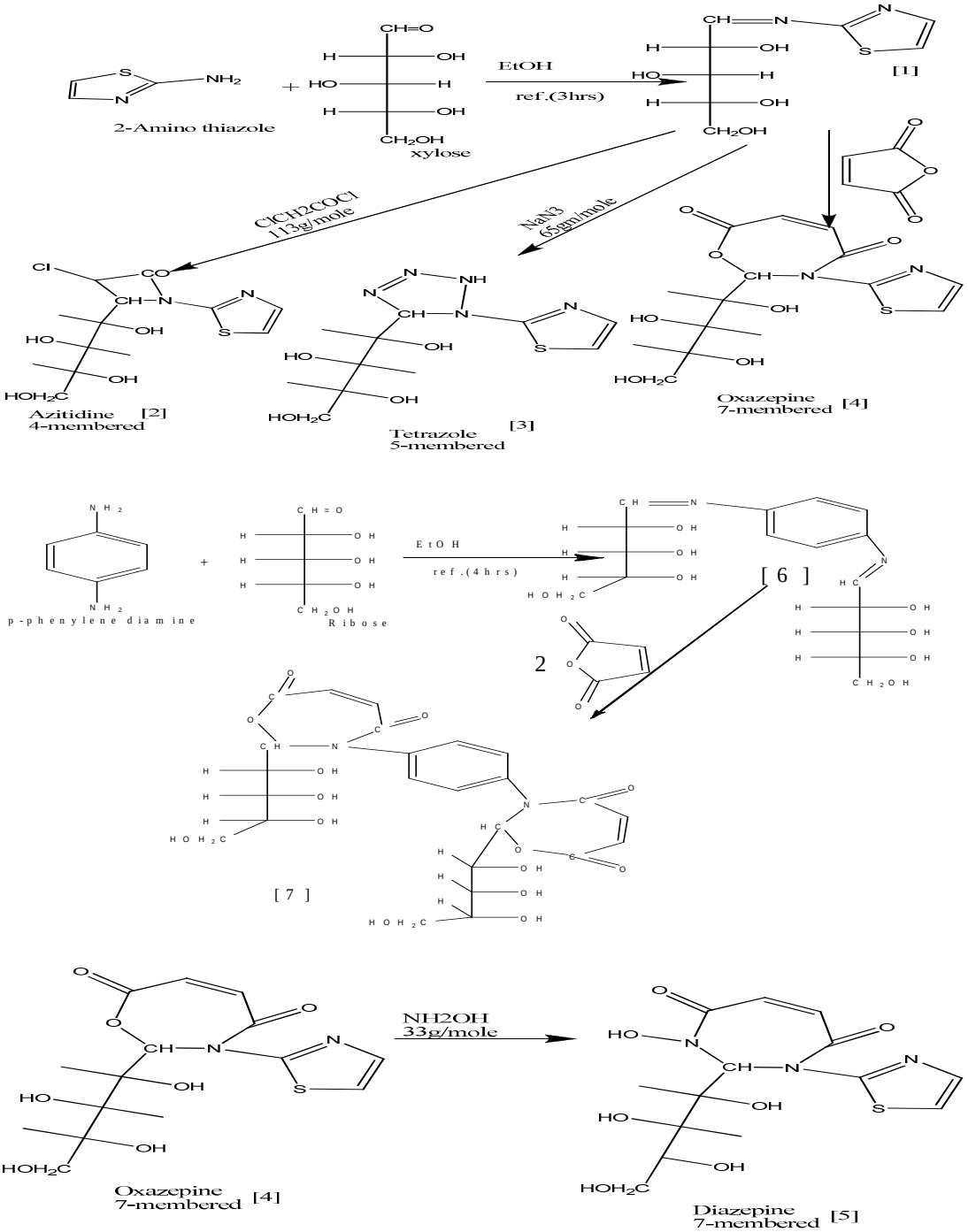
A mixture of (0.1mole,10.8gm) of phenylene diamine with (0.2mole,30gm) of ribose sugar were Heated under reflux for (4hrs) in presence of glacial acetic acid(drops) and absolute ethanol according procedure[12], the precipitation filtered and dried, recrystallized from absolute ethanol To produce 84% of imine-ribose named compound[6].

Synthesis of compound[7]

According procedure[12], amixture of (0.01 mole,3.72gm) of compound[6] with (0.02 mole,1.96gm) of Maleic anhydride were refluxed for (7hrs) with stirre in presence of benzene, after cooled, the Precipitate filtered and dried,recrystallized to produce 86% of seven-membered ring[12,13] of Oxazepine namedcompound[7].

Synthesis of compound[8-10]

To prepare open ring compounds according procedure[12], amixture of (0.01 mole, 5.68gm) of compound[7] with (0.02 mole) from one of{(1.7gm of pipyridine), (1.46gm of diethyl amine), (2.2gm of benzene thiol)} respectively were refluxed for(5hrs) with stirre, after cooled, the precipitate filtered and dried to produce(84,86,83)% of compounds[8-10] respectively.


2-Amino thiazole
xylose
EtOH
ref.(3hrs)
[1]
ClCH2COCl
113g/mole
NaN3
65gm/mole
Azitidine [2]
4-membered
Tetrazole [3]
5-membered
Oxazepine [4]
7-membered
p-phenylene diamine
Ribose
EtOH
ref.(4hrs)
[6]
2
[7]
NH2OH
33g/mole
Oxazepine [4]
7-membered
Diazepine [5]
7-membered

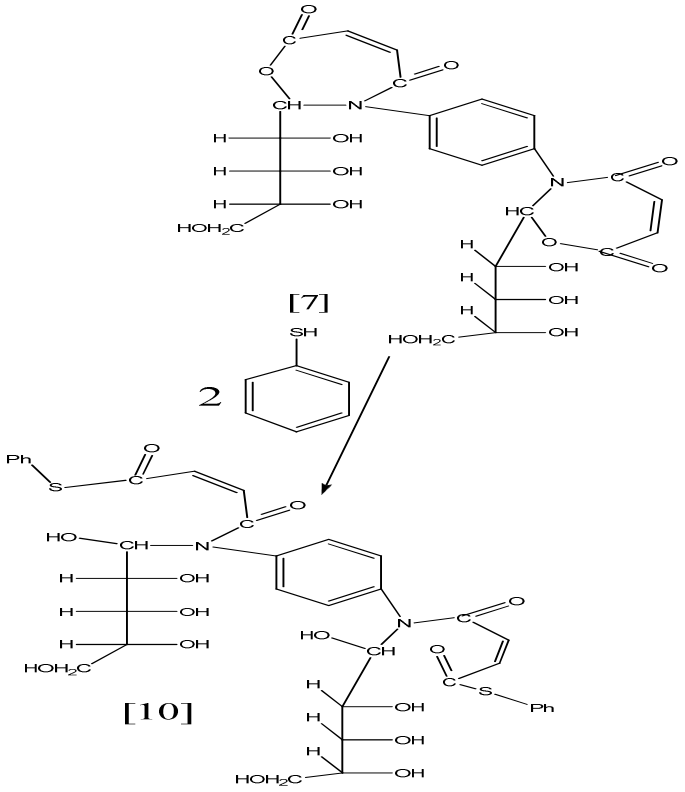


Table (1): (FT-IR)-data(Cm^{-1})of compounds[1-10].

Comp.No	I.R(KBr) (Only Important Groups)
[1]	(CH=N)imine group:1631,(OH)hydroxyl group of sugar:3317
[2]	(CO-N)amide in azetidine cycle:1689,(C-Cl):860,(OH)hydroxyl group of sugar:3385
[3]	(N=N)endocycle:1537,1548,(OH)hydroxyl group of sugar:3462,(NH):3312,(C-N) endocycle:1232
[4]	(CO-O)of oxazepine ring:1728, (CO-N)amide in oxazepine ring:1686, (OH)hydroxyl group of sugar 3501 ,(CH=CH)3086
[5]	(CO-N)amide of oxazepine cycle:1696, (OH):3317, (CH=CH):3091
[6]	(HC=N)imine group:1632, (OH) hydroxyl group of sugar:3398
[7]	(CO-O) of oxazepine ring:1740,(CO-N)amide of oxazepine:1695,(OH)hydroxyl of sugar:3370,(CH=CH):3095
[8]	(CO-N)amide:1688,(OH):3365,(CH=CH):3102,(C-N)endocycle of pipyridine:1290
[9]	(CO-N)amide 1696, (OH):3428,(CH=CH):3091,(C-H)aliphtic:2891,$(N(Et)_2)$:1306
[10]	(CO-N)amide:1690, (OH):3350, (CH=CH):3100 , (C-S):690

Table (2): Physical properties and (C.H.N)-analysis of compounds [1-10]

Comp. No.	M.F	m.p$_{(C°)}$ (+2)	Name of compounds	Calc. / Found		
				C %	H %	N %
[1]	$C_8H_{12}N_2O_4S$	139	N-Thiazolidine xylose imine	41.379 41.221	5.172 5.081	12.06 12.00
[2]	$C_{10}H_{13}N_2O_5SCl$	170	3-Chloro-1-(2-thiazole)-4-(xylose)-azetidine-2-one	38.897 38.701	4.213 4.118	9.076 9.003
[3]	$C_8H_{13}N_5O_4S$	178	1-(2-Thiazole)-5-(xylose)-tetrazole	3.909 34.710	4.727 4.514	25.454 25.398
[4]	$C_{12}H_{14}N_2O_7S$	200	3-(2-Thiazole)-2-(xylose)-4,7-dione-1,3-oxazepine	43.636 43.448	4.242 4.103	8.484 8.319
[5]	$C_{12}H_{15}N_3O_7S$	189	1-Hydroxy-3-(2-thiazole)-2-(xylose)-4,7-dione-1,3-oxazepine	41.739 41.587	4.347 4.207	12.173 12.041
[6]	$C_{16}H_{24}N_2O_8$	156	1,4-phenylene-bis(ribose imine)	51.612 51.383	6.451 6.460	7.526 7.361
[7]	$C_{24}H_{28}N_2O_{14}$	212	1,4-phenylene-bis((2-ribose)-4,7-dione-1,3-oxazepine	50.704 50.409	4.929 4.781	4.929 4.743
[8]	$C_{34}H_{50}N_4O_{14}$	198	1,4-phpnylene-bis(N-(hydroxyl ribose)-N-(4-pipyridine)-2- butene-1,4-dione	55.284 55.113	6.775 6.524	7.588 7.463
[9]	$C_{32}H_{50}N_4O_{14}$	180	1,4-phpnylene-bis(N-(hydroxyl ribose)-N-(4-diethyl amine)-2-butene-1,4-dion	53.781 53.573	7.002 7.008	7.843 7.697
[10]	$C_{36}H_{40}N_2O_{14}S_2$	192	1,4-phpnylene-bis(N-(hydroxyl ribose)-N-(4-benzene sulfide)-2-butene-1,4-dione	54.822 54.70	5.076 5.00	3.553 3.374

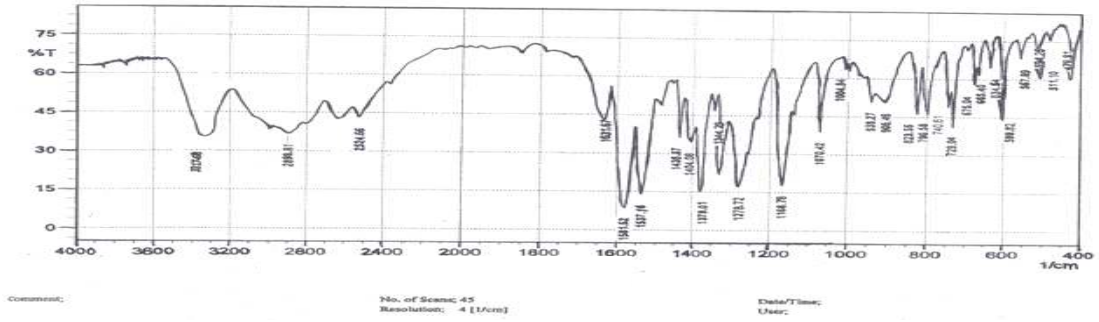


Fig. 1: FT-IR of Compound [1]

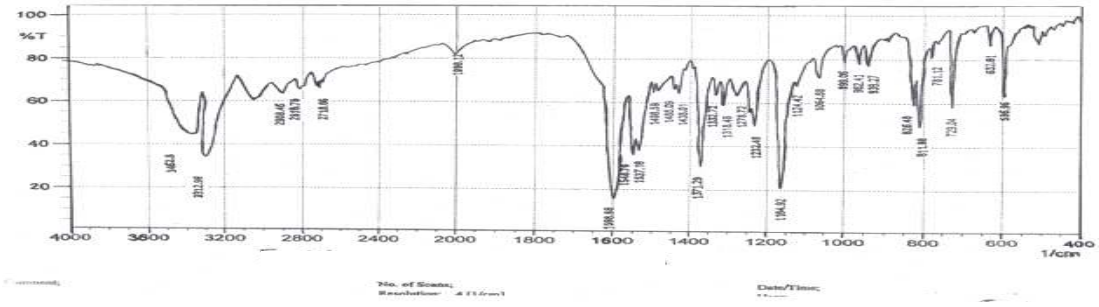


Fig. 2: FT-IR of Compound [3]

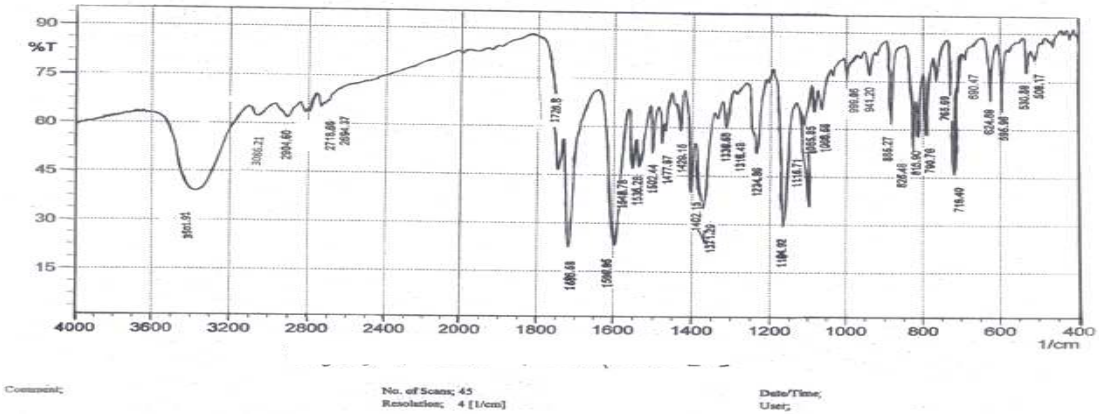


Fig. 3: FT-IR of Compound [4]

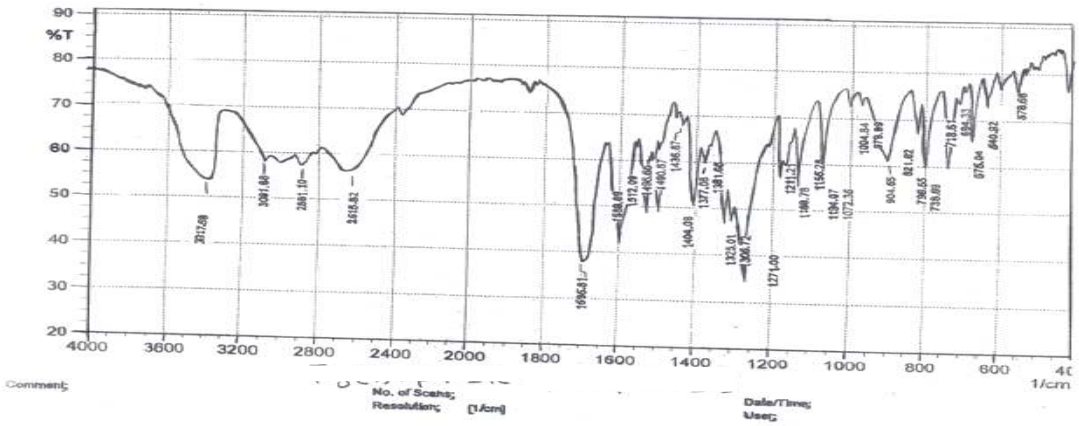


Fig. 4: FT-IR of Compound [5]

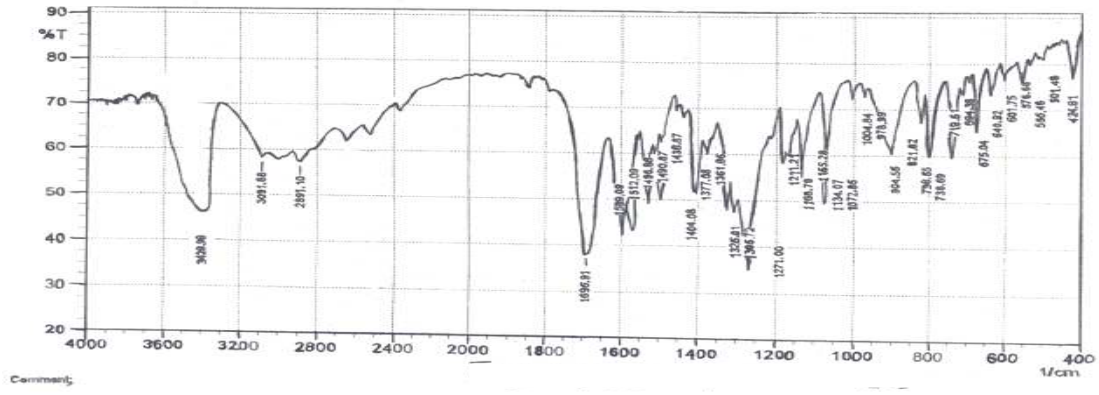


Fig. 5: FT-IR of Compound [9]

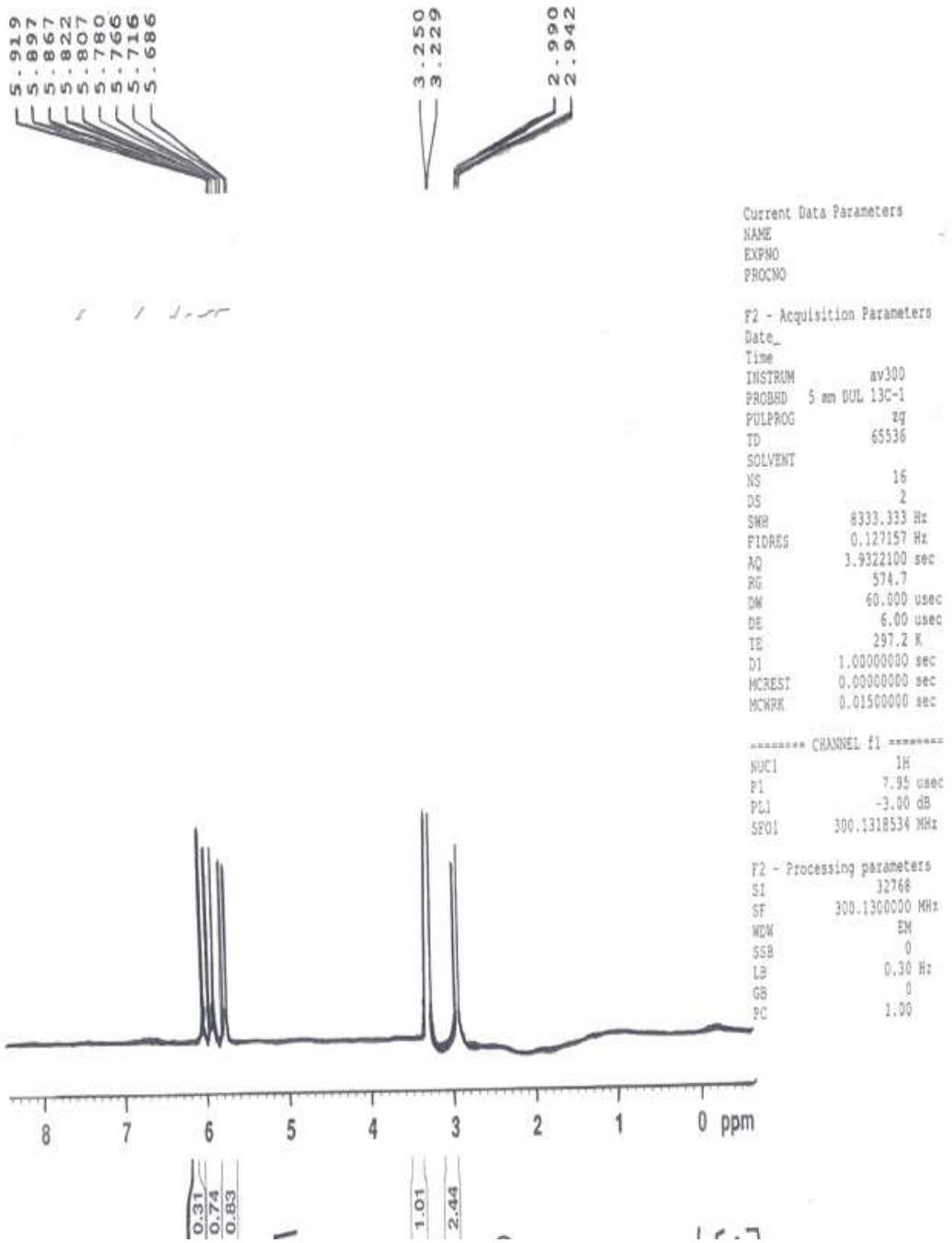


Fig. 7: H-N MR of Compound [2]

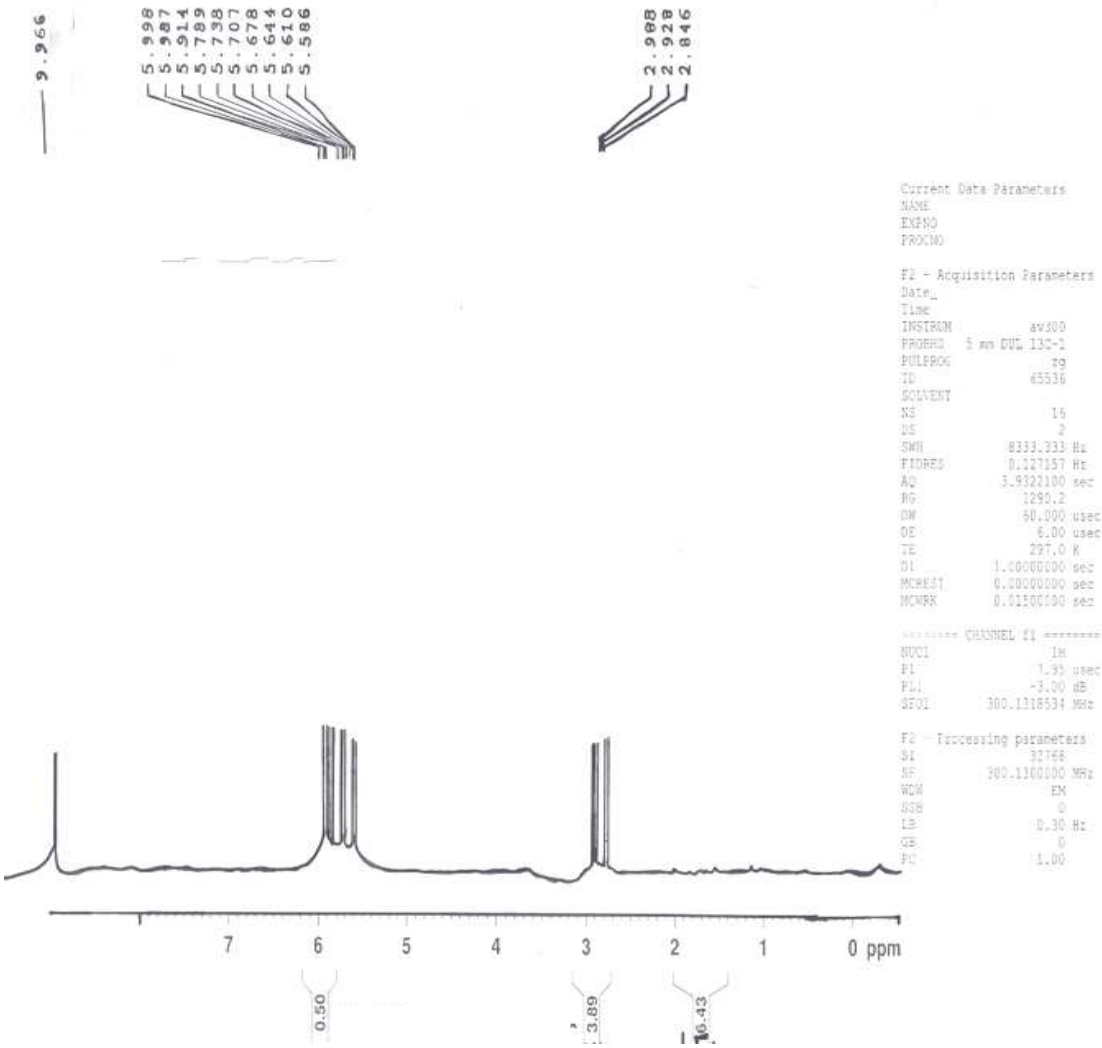


Fig. 8: H-N MR of Compound [4]

RESULTS AND DISCUSSIONS

The mechanism of formatted compounds[1-10] proceed through reactions between Mono saccharide xylose or ribose with primary amins to produce Imine compound of xylose sugar Which reacts with (sodium azide,chloro acetyl chloride,maleic anhydride,primary amine)to Produce fused cyclic compounds[2-5],while the imine comp.of Ribose sugar reacts with maleic anhydride to give oxazepine compound[7] which reacts with(secondary amines,thiol compound) to produce open cyclic compounds[8-10] such as amide.The prepared compounds[1-10] have been

characterized by their melting points and Spectroscopic methods (FT-IR, H-NMR, C.H.N analysis).

The FT-IR- spectrum,showed an absorption bands at (1631,1632)cm^{-1}due to (CH=N) Imine Groups[12,13] in compounds [1,6] respectively, which disappeared and other bands appeared such as{(1689$cm^{-1}$of CO-N amide)[6,12],(860 $cm^{-1}$of C-Cl)}due to azetidine cycle in compound[2],bands at {(1537$cm^{-1}$of –N=N-),(3312 $cm^{-1}$of NH)[3] (1232cm^{-1}ofC-N endocycle)}due to tetrazole cycle of compound[3], bands at {(1728$Cm^{-1}$of CO-O), (1686$cm^{-1}$of CO-N amide)} due to oxazepine cycle[12,13] in compound[4], bands at (1696 $Cm^{-1}$of CO-N amide)[6,12] due to diazepine cycle in compound[5],bands at {(1740 cm^{-1} of CO-O), (1695$cm^{-1}$of CO-N amide)} due to oxazepine cycle in compound[7],bands{(1688$cm^{-1}$of CO-N amide) (1290cm^{-1} of C-N endocycle of pipyridine)}.

In amide of compound[8],bands at{(1696$Cm^{-1}$of CO-N),(2891Cm^{-1}ofC-H aliphatic),(1306Cm^{-1} of $N(Et)_2$ due to amide in compound[9],bands at {(1690 cm^{-1} of CO-N amide, (690 cm^{-1} of C-S)}[6] due to sulfide in compound[10], and other functional groups shown in table(1) and figurs(1-5).

The H-NMR- spectrum showed signal at ∂(8.89) due to (CH=N)proton of imine group in Compound[1],which disappeared and other signal appeared at {(2.99 of CH-Cl),(3.25 of CH-N)}due to azetidine cycle in compound[2]; signals at {(9.96 of N-CH-O)[12,13], (2.84,2.98 of CH=CH)} due to oxazepine ring in compound[4]; signals at{(3.76 of N-CH-N),(4.30 of N-OH), (2.73,2.91 of CH=CH)} of diazepine ring in compound[5]; signals at {(3.38 of $N(Et)_2$),(2.27,2.72 of CH=CH)} in compound[9], and other signals of functional groups[14-16] show in the following, table (2) and figures (6-9). The (C.H.N)analysis and melting points, it was found from compard the calculated data with experimentally data of these compounds,the results were very good. The data of analysis, M.F and melting points are listed in table (3).

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